Fabrication of Diamond Membranes for MEMS using Reactive Ion Etching of Silicon

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Abstract

Polycrystalline diamond thin film has been grown on a silicon substrate using high pressure microwave plasma-assisted chemical vapor deposition from a gas mixture of methane and hydrogen at a substrate temperature of 950°C. A simple process flow has been developed to fabricate optically transparent polycrystalline synthetic diamond membranes/windows employing reactive ion etching (RIE) of single crystal silicon substrate using electron beam evaporated aluminum thin film mask pattern formed by photolithography. Scanning electron microscopy has been used to study morphology of as-grown diamond thin films.

Key words: **diamond, membrane, reactive ion etching, etching of silicon**

INTRODUCTION

Growth of polycrystalline diamond thin films using high pressure and high temperature microwave plasma-assisted chemical vapor deposition has received significant interest in recent years, since it has unique chemical and physical properties for potential microelectromechanical (MEMS) applications. Diamond growth with microwave plasma processes at pressures 10 - 760 Torr has been achieved by several groups. [1-6] It is necessary to scratch damage the polished silicon substrate using diamond paste to enhance the nucleation density of diamond to achieve a pinhole-free continuous layer of diamond.[7] In order to facilitate application of diamond films for MEMS, a technique to fabricate diamond microstructures or mechanical components are desirable.

MEMS is a microelectronics fabrication approach to miniaturize the electromechanical sensor devices and integrate with the integrated circuit (IC) fabrication processes. MEMS devices may have a significant application in automotive, displays, printers, fluid thrusters, analytical instruments, communications, biomedical, and aerospace industry. Operation, reliability, sensitivity, and stability of smaller, lighter, and cheaper MEMS devices is very critical in any chosen application particularly under extreme shock and ambient temperature conditions. These MEMS devices should be built in along with other semiconductor devices using integrated semiconductor fabrication technologies. New material such as silicon carbide and diamond and their process technology to fabricate the MEMS devices is necessary for high temperature applications where silicon may not be applicable at temperature more than 150°C. MEMS devices may have a use to monitor a wide variety of parameters such as temperature, accelerations, flow rates, pressures, vibrations, surface wear rates, fluid contaminants, position sensing, etc.

A novel method was reported earlier to selectively deposit polycrystalline CVD diamond on a silicon surface and further demonstrated the fabrication of diamond microstructures such as membranes (four sides supported), cantilever beams (single side supported), and bridges (double side supported) using anisotropic chemical etching of silicon. [8] This technique involves the protection of silicon surface by using low pressure chemical vapor deposited silicon nitride (LPCVD) from attacking the front side of the silicon wafer by hot KOH chemical solutions. The etching rate of silicon is ~1 μm/minute in KOH solution at 60°C. The method is relatively cumbersome such as cleaning the wax and any other masking materials which are used to protect the silicon surface. The etching of silicon substrate in KOH solution is anisotropic in nature. Salvadori et al., [9] have reported the fabrication of free-standing diamond membranes using 'O' ring instead of mask materials. This approach certainly does not allow the defined dimensions of the microstructures for sensor applications. Noguchi et al., [10] have reported the fabrication of diamond membranes using KOH solutions for x-ray lithography masks. Deng and Ko [11] have shown that the diamond-like-carbon (DLC) may be used as friction-reducing coating materials for MEMS applications. Houston et al., [12] have reported the use of diamond-like-carbon film to passivate the silicon surfaces to reduce stiction in microelectromechanical devices. Stiction is one of the most difficult problem that is being experienced by MEMS research community. Aslam and Schulz [13] have reported the selective diamond nucleation technology employing electroplated chromium as a mold during diamond deposition for microelectromechanical applications.

In this paper, we describe a process flow combined with conventional photolithography and reactive ion etching of silicon to fabricate diamond membranes for microelectromechanical device applications. We have used a well-established reactive ion high etching rate of silicon to fabricate diamond membranes for the first time to enhance the yield of MEMS devices. This approach is three to six times faster than the hot KOH anisotropic etching process. This process may also be used to fabricate diamond microstructures such as cantilever beams and bridges for microsensor and MEMS applications.

EXPERIMENTAL

A commercially available microwave plasma-assisted CVD system (ASTeX, Woburn, MA) was used in our experiments to grow polycrystalline diamond thin films. The schematic diagram of the polycrystalline diamond growth system and growth process details are described in earlier publications.[7, 8, 14, 15, 16]

Starting substrates were mirror-smooth finished n- or p-type, (100) oriented single crystal silicon wafers with a resistivity of <20 ohm-cm. The wafers were cleaned in acetone, methanol, deionized (DI) water, and dried with nitrogen gas.

A continuous film of polycrystalline diamond was usually obtained after 15 - 20 h of growth. Growth rate of microwave plasma CVD diamond is typically 1 μm/hour. The typical deposition parameters were as follows: substrate temperature = 950 °C, methane flow rate = 3.6 sccm, hydrogen flow rate = 500 sccm, deposition pressure = 45 Torr (5.985 x 10^2 kpascals), forward power = 1200 W, reflected power = 34 W, and the percent of methane in hydrogen = 0.71%. Films grown under these conditions have been analyzed earlier by Raman spectroscopy and x-ray diffraction and they were confirmed to be diamond.[16]

The aluminum was deposited on the backside of the CVD diamond grown silicon substrate by electron beam evaporation technique. The aluminum was then photolithographically patterned and etched using commercially available phosphoric acid and acetic acid etching (PAE) solution.

The photoresist was cleaned with acetone, rinsed with methanol, DI water, and finally dried with nitrogen gas. The evaporation parameters employed during deposition process are: base pressure = 5 x 10^{-6} Torr (6.65 x 10^{-4} kpascals), voltage = 6 - 9 kV, current = 0.3 - 0.4 amperes, deposition rate = 0.06 μm/minute, thickness of aluminum film = 0.38 μm.

Reactive ion etching (Drytek Plasma Systems Quad 480 series) has been employed to etch silicon to fabricate diamond membranes. The parameters employed in this etching process are as follows: SF_6 flow rate = 10 standard cubic centimeter per minute (sccm) (0.01 standard liters per minute, slm), O_2 flow rate = 10 sccm (or 0.01 slm), RF forward power = 295 watts, Reflected RF power = 0 watts, and DC bias = 22 watts. The total silicon wafer thickness was 630 μm in this study. The total etching time for the complete silicon wafer was 2 hours 45 minutes. The etching rate was found to be ~3.6 μm/minute. The etching rate of silicon depends on the area of the silicon surface exposed to the RIE plasma. This dependence may be called as silicon loading effect. Qualitatively, the etching rate of silicon is higher if the exposed silicon surface area is smaller. Figure 1 shows the schematic process flow steps to fabricate membranes of chemical vapor deposited polycrystalline diamond films grown over a single crystal silicon substrate.

RESULTS AND DISCUSSION

Figure 2(a) is a scanning electron micrograph of a diamond membrane over a Si substrate using process flow steps including reactive ion etching steps shown in Fig. 1. Figure 2(b) is a scanning electron micrograph of the morphology of polycrystalline diamond on the backside of the diamond membrane. The morphology on the backside of the membrane is smooth when compared with the top of the diamond film (Fig 3). The silicon etch rate was found to be in the range of 3.6 μm/minute. The exposed area of silicon during the RIE process is ~1.01 cm^2. The aluminum was used as a mask material to etch the silicon in a desired area. Undercutting or underetching of silicon was observed when aluminum was used as a mask material during RIE process. The undercutting was observed to be in the range of 578 μm over an etch duration of 165 minutes. The silicon undercutting rate was found to be 3.5 μm/minute which was apparently equal to the vertical etch rate of silicon during the same RIE process. It was quite surprising to note that the etch rate was equal to the undercutting rate of silicon substrate. Therefore, it is necessary to find a mask material to etch silicon with a minimum undercutting rate when compared with the etch rate of silicon substrate vertically. Aspect ratio should be as high as possible to fabricate microstructures for MEMS applications. This micrograph shows the feasibility of fabrication of diamond membranes for eventual MEMS applications.

Figure 3(a) is a SEM micrograph showing a typical morphology of as-grown diamond film on a silicon substrate. The diamond morphology was found to be poor compared with the morphologies that we have reported earlier in the literature using the same CVD apparatus.[7, 8, 14 - 16] This film in the membrane was confirmed to be diamond using EDAX chemical analysis. If it were to be graphite it would have etched during RIE process that contains oxygen as a reactant in the plasma. We did not analyze this film using Raman analysis since this technique is not available for analysis at the present time. Our objective in this paper is not to find out the morphology of as-grown film but to propose a method to fabricate diamond membranes and other microstructures using reactive ion etching process. We have analyzed diamond films deposited using the same system and the results were reported earlier.[7, 8, 14 - 16] Figure 3b shows the scanning electron micrographs of the top side of diamond membranes. Figure 3c shows the magnified view of the scanning electron micrograph shown in Fig. 3b. It is clear from the Figs 3b and 3c that the diamond membranes are buckling up due to the stress that had built-up during the diamond deposition performed at 950±25°C. We have not analyzed the amount of stress that had built-up in the diamond film in this study. We will be fabricating the diamond membranes of various sizes and further evaluate quantitatively the amount of stress that is present in the diamond films. Our future work will address some of these issues for a successful use of diamond for MEMS applications.

Figure 4 shows the etch depth of silicon substrate as a function of etch duration. Reactive ion etching was performed under similar conditions as performed in the Fig. 2. The surface area of the exposed silicon surface is about 1.44 cm^2 which is higher than the sample area employed the experimental results shown in Fig. 2. The etching rate of silicon is approximately 2.5 μm/minute which is lower than the etch rate of 3.5 μm (surface area = 1.01 cm^2) reported in the Fig. 2. Therefore, it may qualitatively be concluded that the etch rate of silicon decreases as the exposed area of the silicon for the plasma increases.

SUMMARY

A process has been developed and demonstrated to fabricate polycrystalline diamond membranes on a silicon substrate using reactive ion etching of silicon. Bulk micromachining of silicon has been performed using KOH solution in the literature to fabricate diamond membrane. The etching rate of silicon in hot KOH solution is ~1 μm/min besides it is anisotropic in nature. It has been quite a routine process such as bulk micromachining the silicon for microelectromechanical device applications. This approach has allowed the MEMS community to fabricate sensor devices with high yields in shorter duration by using RIE process. Therefore, we have attempted to fabricate diamond membranes using silicon bulk micromachining process using dry reactive ion etching process. In silicon MEMS devices it is needed an etch stop layer to fabricate microstructures. RIE plasma will not attack the diamond which will be an advantage for diamond MEMS applications. This approach can be used to fabricate any diamond microstructures successfully for MEMS applications. The undercutting rate was found to be in the range of 3.5μm/min which is considered to substantially higher than we have expected. The diamond film was found to be under compressive stress once the diamond film is relieved from the silicon substrate after reactive ion etching of silicon. We have demonstrated the etching rate of silicon increases as the surface area of the exposed silicon varies in the plasma decreases.

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Figure Captions

1. Schematic diagram of the process flow steps to fabricate CVD diamond membrane.
2. Scanning electron micrographs of (a) backside of diamond membrane and (b) typical morphology of the backside of the fabricated diamond membrane using RIE etching process,
3. Scanning electron micrographs of (a) top side of diamond membranes, (b) magnified view of "a", and (c) typical morphology of the diamond film in the fabricated diamond membrane area.
4. Etch depth in silicon substrate vs etch duration using reactive ion etching process in SF_4 and O_2 reaction mixture.

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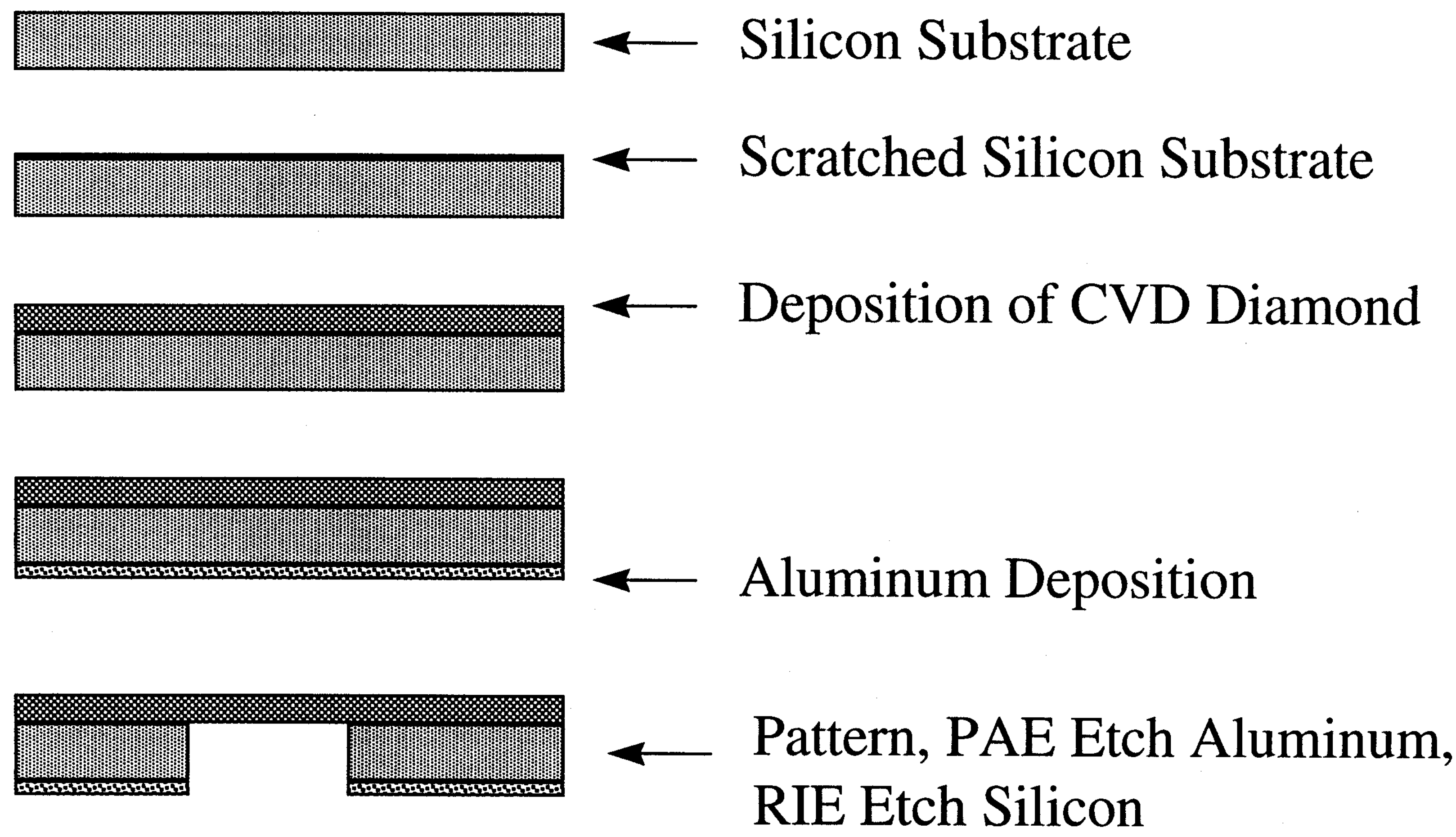


Figure 1: Ramesham et al.,

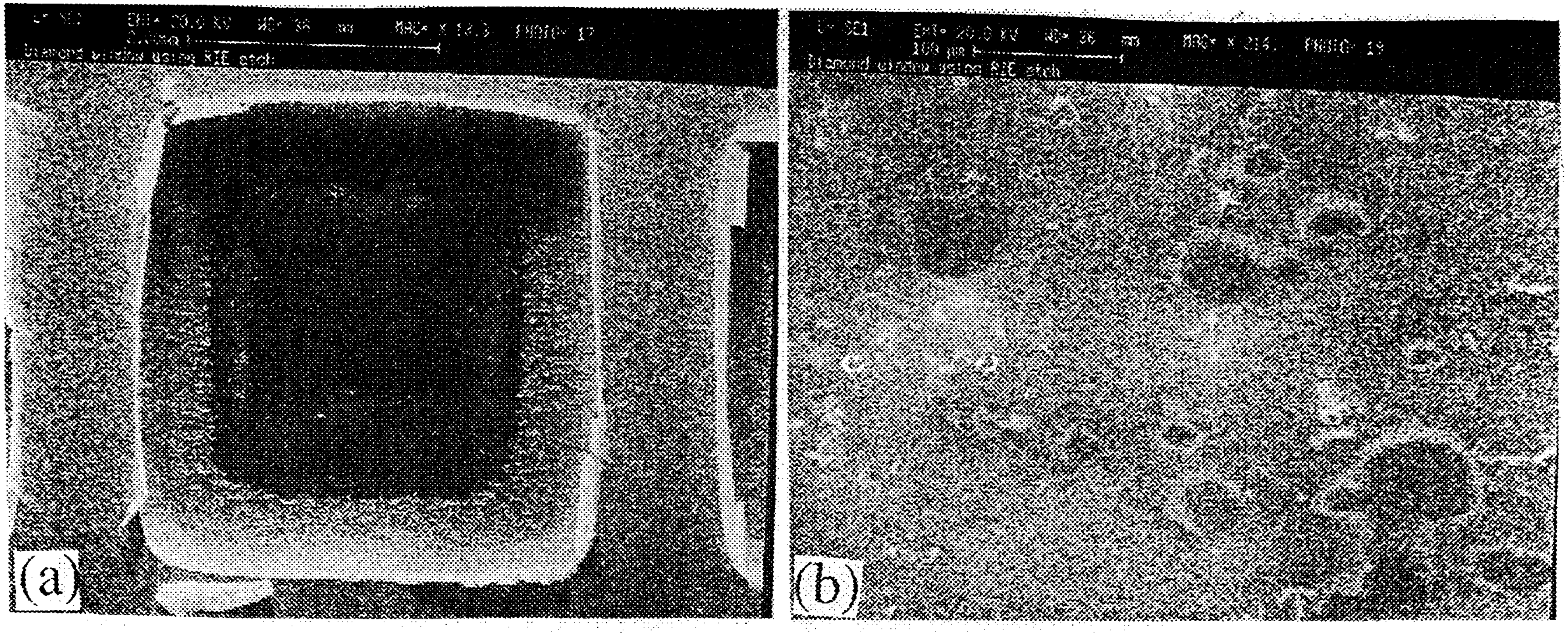
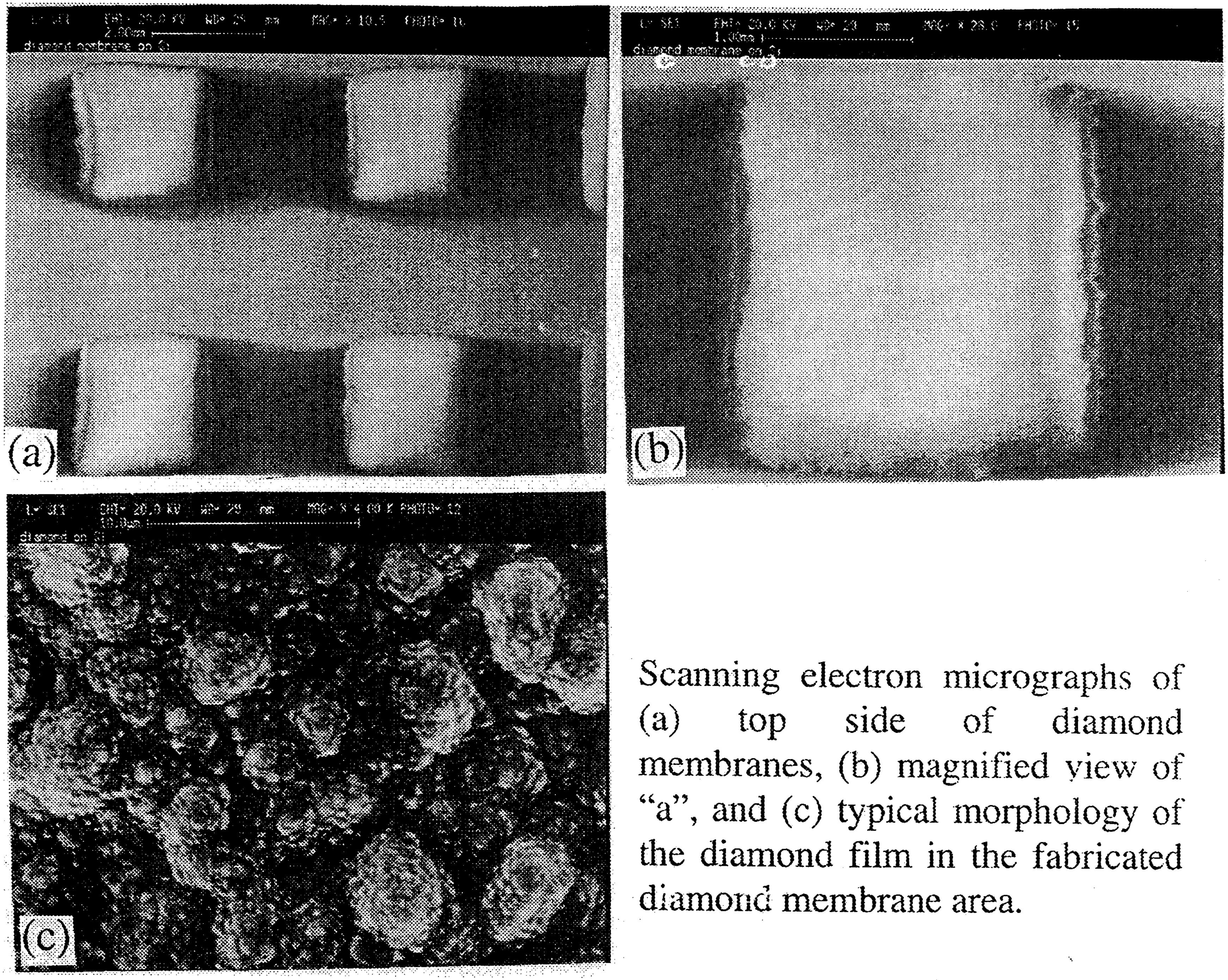


Figure 2: Ramesham etal.



Scanning electron micrographs of (a) top side of diamond membranes, (b) magnified view of "a", and (c) typical morphology of the diamond film in the fabricated diamond membrane area.

Figure 3: Ramesham et al.

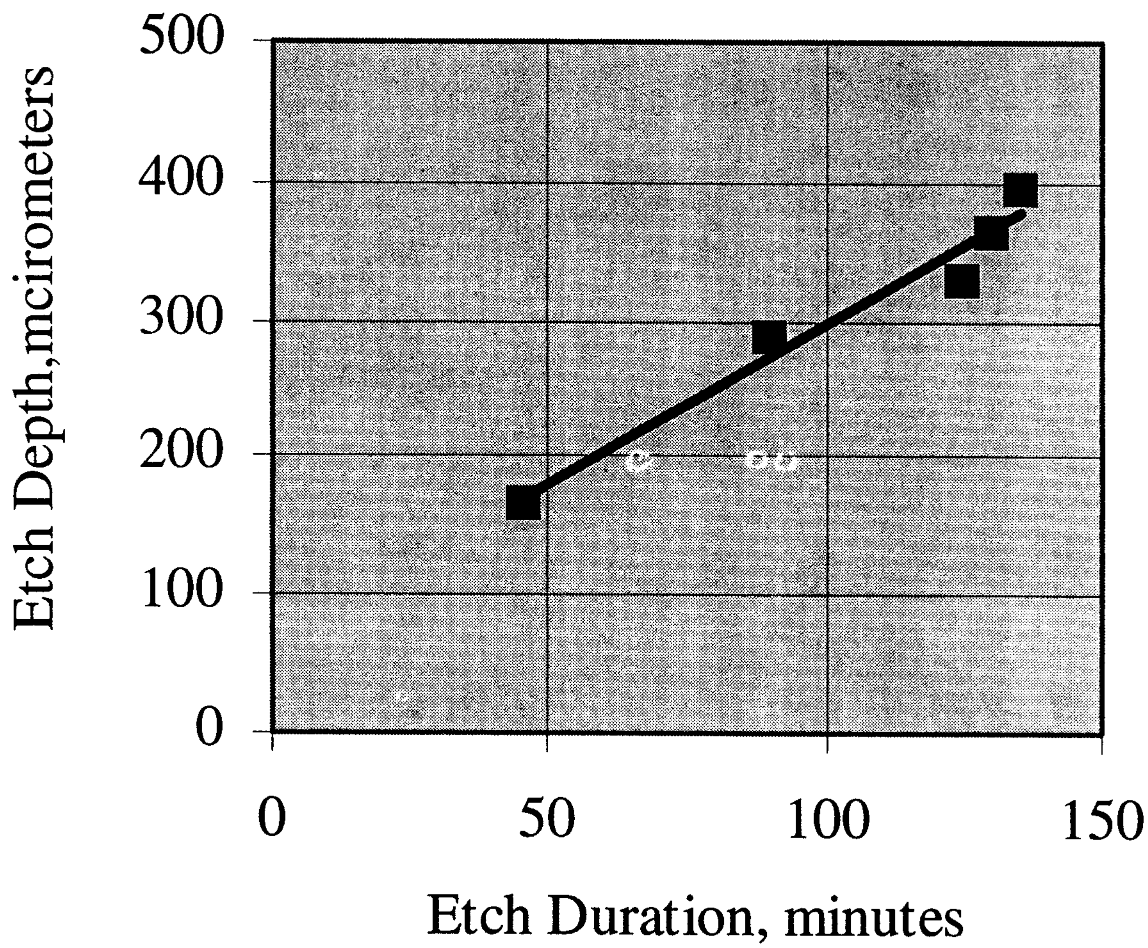


Figure 4: Ramesham et al.,